

Certificate of Analysis

Standard Reference Material® 2724b

Sulfur in Diesel Fuel Oil

This Standard Reference Material (SRM) is intended for use in the evaluation of methods and the calibration of instruments used in the determination of total sulfur in fuel oils or materials of similar matrix. SRM 2724b is a commercial "No. 2-D" distillate fuel oil as defined by ASTM D 975-96a Standard Specification for Diesel Fuel Oils [1]. A unit of SRM 2724b consists of 10 amber ampoules, each containing approximately 10 mL of diesel fuel.

Certified Value: The certified sulfur content provided in Table 1 is based on analyses by isotope dilution thermal ionization mass spectrometry (ID-TIMS) [2]. Homogeneity testing was performed using x-ray fluorescence spectrometry. The uncertainty in the certified value is expressed as an expanded uncertainty and is calculated according to the method in the ISO Guide [3]. The expanded uncertainty is based on a 95 % prediction interval.

Table 1. Certified Value (mass fraction)

Sulfur: $0.04265 \% \pm 0.00057 \%$

Information Values: Information values are provided in Table 2 for additional properties of SRM 2724b. The results are not certified and no uncertainty is reported. The results are given to provide additional information on the matrix.

Expiration of Certification: The certification of this SRM is valid until **31 December 2008**, within the uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in the certificate (see Instructions for Use). However, the certification will be nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: This material is considered to be stable during the period of certification. NIST will monitor this material and will report any significant changes in certification to the purchaser. Return of the attached registration card will facilitate notification.

The overall direction and coordination of the technical measurements leading to certification of this SRM were performed by J.D. Fassett of the NIST Analytical Chemistry Division. Analytical measurements were performed by W.R. Kelly, R.D. Vocke, A.F. Marlow, J.R. Sieber, and J.L. Mann of the NIST Analytical Chemistry Division.

Statistical consultation for this SRM was provided by K.R. Eberhardt of the NIST Statistical Engineering Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald.

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Gaithersburg, MD 20899 Certificate Issue Date: 28 March 2001 See Certificate Revision History on Last Page Nancy M. Trahey, Chief Standard Reference Materials Program

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Instructions for Use: Each SRM ampoule should only be opened for the minimum time required to dispense the material. Once an ampoule is opened, the material must be used within a period of 8 h to avoid a significant change in the sulfur content. To relate analytical determinations to the certified value in this Certificate of Analysis, a minimum sample mass of 150 mg should be used. The unopened ampoules should be stored under normal laboratory conditions away from direct sunlight.

Table 2. Information Values for Selected Properties

Physical Property Test*	ASTM Standard Used	Result
Density @ 15 °C @ 60 °F	D 1250-80 (1990) D 287-92 (1995)	822.3 kg/m³ 40.5 API
Flash Point	D 93-94	73.0 °C
Pour Point	D 97-93	- 18.0 °C
Heat of Combustion, Gross	D 240-92 [?] 1	45.99 MJ/kg (19 771 Btu/lb)
Kinematic Viscosity @ 40 °C	D 445-94 [?] 1	$2.344 \times 10^{-6} \mathrm{m^2/s} (2.344 \mathrm{cSt})$
Color	D 1500-96	L 0.5
Oxidation Stability	D 2274-94	<0.1 mg/100 mL Filterable <0.1 mg/100 mL Adherent <0.1 mg/100 mL Total
Carbon	D 5291-92	85.5 %
Hydrogen	D 5291-92	11.9 %

^{*} These properties were determined by a commercial firm under contract to NIST using ASTM methods. The results are **NOT** certified and are provided as additional information on the matrix.

ASTM Standards

D 93-94	Standard Test Methods for Flash Point by Pensky-Martens Closed Cup Tester
D 97-93	Standard Test Method for Pour Point of Petroleum Products
D 240-92 ^{?1}	Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb
	Calorimeter
D 287-92 (1995)	Standard Test Method for API Gravity of Crude Petroleum and Petroleum Products
	(Hydrometer Method)
D 445-94 ^{?1}	Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (the
	Calculation of Dynamic Viscosity)
D 1250-80 (1990) ^{?1}	Standard Guide for Petroleum Measurement Tables
D 1500-96	Standard Test Method for ASTM Color of Petroleum Products (ASTM Color Scale)
D 2274-94	Standard Test Method for Oxidation Stability of Distillate Fuel Oil (Accelerated Method)
D 5291-92	Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen
	in Petroleum Products and Lubricants

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^{?1} Indicates that only editorial changes were made to the previous issuance of the ASTM standard.

REFERENCES

- [1] ASTM D 975-96a, "Standard Specification for Diesel Fuel Oils," *Annual Book of ASTM Standards* **05.01**, West Conshohocken, PA, (1997).
- [2] Kelly, W.R., Paulsen, P.J., Murphy, K.E., Vocke, R.D., Jr., and Chen, L.-T., "Determination of Sulfur in Fossil Fuels by Isotope Dilution Thermal Ionization Mass Spectrometry," Anal. Chem. **66**, pp. 2505-2513, (1994).
- [3] Guide to the Expression of Uncertainty in Measurement, ISBN 92-67-10188-9, lst Ed. ISO, Geneva, Switzerland, (1993); see also Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington DC, (1994); (available at http://physics.nist.gov/Pubs/).

Certificate Revision Date History: 28 March 2001 (Certified sulfur value and uncertainty revised); 09 September 1999 (Original version).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.

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